

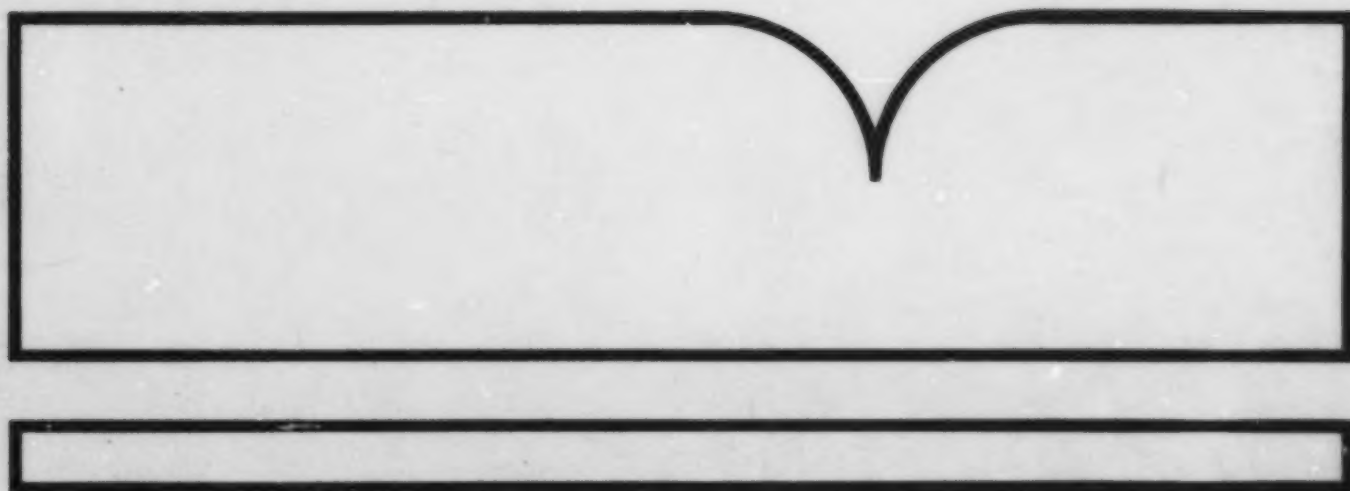
The Feasibility of Identifying  
Mystery Oil Spills

Maine Dept. of Environmental Protection  
Augusta

Prepared for

Municipal Environmental Research Lab.  
Cincinnati, OH

Apr 81



U.S. Department of Commerce  
National Technical Information Service

**NTIS**

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| Project Officer:<br>Michael Gruenfeld<br>(201) 321-6625                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                                                                                                    |                                                |
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THE FEASIBILITY OF IDENTIFYING  
MYSTERY OIL SPILLS

by

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U.S. ENVIRONMENTAL PROTECTION AGENCY  
CINCINNATI, OHIO 45268

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FOREWORD

The U.S. Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimonies to the deterioration of our natural environment. The complexity of that environment and the interplay of its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution; it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems to prevent, treat, and manage wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, to preserve and treat public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research and provides a most vital communications link between the researcher and the user community.

This report describes and compares several passive tagging techniques for identifying the origin of mystery oil spills. The effectiveness of the techniques is evaluated by assessing their viability for enforcement of relevant provisions of Maine's Oil Conveyance Law. This study will be of interest to all those concerned with oil passive tagging (fingerprinting) methods of chemical analysis, used for identifying the source of discharge of petroleum oil spills.

Francis T. Mayo, Director  
Municipal Environmental Research  
Laboratory

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ABSTRACT

Several off-the-shelf passive tagging techniques for identifying the origin of mystery oil spills were evaluated to determine the viability of enforcement provisions of Maine's Oil Conveyance Law. Duplicating the operating conditions experienced during every-day marine terminals activities, ship's samples of all crude oil shipments received during 15 day intervals in Portland (Maine) Harbor were collected.

Selected samples from those collected during a test interval were subjected to artificial spills in 500 gallon tanks exposed to outdoor, sea-side ambient conditions experienced in Portland, Maine. Subsamples of each artificial spill were taken during the 15 day test interval to determine the effect of weathering. Several test intervals were conducted over a period of two years to observe seasonal effects.

Attempts were made to match weathered unknowns to collections of known oils containing the "mystery" oil. Several passive tagging techniques were used such as the comparison of high resolution and low resolution gas chromatography, vanadium-nickel ratios, sulfur nitrogen ratios, and infrared spectra. Novel procedures to facilitate numerical comparisons were developed. Successes, failures, and limitations of the passive tagging methods are discussed.

The report was submitted in fulfillment of contract Number R801006 by the Maine Department of Environmental Protection under the sponsorship of the U. S. Environmental Protection Agency. This report covers a period from 1972 to 1975 and work was completed as of 1977.

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The authors also wish to thank the Commanding Officer, Brunswick Naval Air Station, U. S. Navy, for the use of their S. Harpswell dock facilities.

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SECTION 1

INTRODUCTION

Maine's landmark Oil Conveyance Law of 1970 was established to protect the public health and welfare from the hazards of the conveyance of oil and to ensure the rapid cleanup of spills. To enable recovery of the cleanup costs of mystery oil spills, certain regulations were promulgated. Namely, all marine terminal operators onloading or unloading shipments of petroleum products must take a sample of each product and retain it for a period of 15 days under specified conditions. By the successful use of proper tagging techniques it should be possible to match a mystery oil spill to one of the reference samples retained by the technical operator. The liable operator can then be assessed the cost of cleanup.

The purpose of this study is to evaluate existing passive tagging techniques readily available to a regulatory agency such as Maine's Department of Environmental Protection. Furthermore, the study attempts to show if the passive tagging techniques evaluated under every-day operating conditions experienced by Maine's Oil Conveyance Law constitute a reliable, effective enforcement tool to determine the origin of mystery oil spills.

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SECTION 2

CONCLUSIONS

1. A passive tagging technique employing a numerical comparison of heptadecane, pristane, octadecane, and phytane ratios from high resolution gas chromatograms plus comparison of Ni, Vanadium, sulfur, and Nitrogen ratios is highly successful at matching spilled crude oil to a reference collection of crude oils.
2. The best passive tagging technique is moderately successful at matching a spilled crude oil to the correct tanker load where multiple loads from the same oil field exist.
3. Oil weathered more than two weeks may begin to experience degradation in the chromatographic region used for passive tagging.
4. The visual comparison of low and high resolution gas chromatograms is of limited usefulness as a passive tagging tool.
5. Fifteen (15) meter SCOT OVI columns are suitable for resolving hexadecane, pristane, octadecane, and phytane and possess good intercolumn and intracolumn reproducibility.

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SECTION 3

RECOMMENDATIONS

1. Additional work must be performed to determine the impact of weathering beyond two weeks on passive tagging techniques.
2. An evaluation of the distribution of the chemical composition in a tanker and the effect of that distribution on passive tagging should be conducted.
3. Passive tagging techniques should be verified using petroleum distillates and refined products.

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SECTION 4

FIELD STUDIES

SAMPLING

Maine law (MRSA Title 38, Chapter 3, Section 546) enables the promulgation of rules and regulations necessary for its enforcement. Pursuant to that end, each marine transporter of petroleum products must collect a mid-ship sample of each cargo and store it for a period of 15 days. See Table 1.

In order to duplicate reality as closely as possible, project sampling procedures were very similar to those required by regulation. That is, a mid-column sample from a randomly selected vessel hold was collected in a brass "Thief" prewashed in hexane. The type of sampling device used is shown in Illustration 1. Obviously, the use of non-sparking materials was absolutely essential. Although perhaps not of scientific interest, nevertheless the act of boarding a foreign vessel for sampling purposes proved to be a major logistical-administrative effort. The entire procedure for ship sampling is shown in Table 2.

The mid-column sample was the one universally collected throughout the course of the study under the assumption that it would be the most representative of the cargo. This constraint on sampling was necessary because the objective of the project was to study passive tagging techniques, not the distribution of hydrocarbons within an oil cargo hold. However, several spot checks of samples collected near the top and bottom of the oil column were made in the course of the study. These results are discussed later.

Freshly collected samples were transferred to one pint wide mouthed glass bottles prewashed with hexane. The tops were lined with aluminum foil. Samples were then transferred to the TRIGOM laboratory facilities in South Portland for temporary storage prior to distribution to the Bowdoin laboratory for analysis or to field sites for ambient weathering.

WEATHERING FACILITIES

The ambient weathering site was chosen at the U.S. Navy oil depot and docking facilities located at South Harpswell. See Illustration 2. The site was chosen for logistical reasons (proximity to the laboratory and sampling point) as well as for security purposes (the depot area is secured with fencing and is located in a remote area). The possibility of experimental contamination during depot oil handling operations was acknowledged but thought

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TABLE 1. SAMPLING AND STORAGE REGULATION FOR REFERENCE OIL SAMPLES

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REGULATIONS

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DEPARTMENT OF ENVIRONMENTAL PROTECTION

BUREAU OF WATER QUALITY CONTROL

EFFECTIVE DATE OCTOBER 21, 1971

AMENDED: JULY 12, 1973

AMENDED: JANUARY 15, 1974

AMENDED: OCTOBER 1, 1974

AMENDED: FEBRUARY 8, 1978

OIL PREVENTION AND

POLLUTION CONTROL REGULATIONS

601.1 - 619.

603.1 Transfer Operations for Petroleum and Petroleum Products

m. Sample Collection

No terminal operator shall transfer or cause to be transferred or consent to the transfer of any bulk oil from any vessel to a land based oil terminal facility or from a land based oil terminal facility to any oil carrying vessel or from one vessel to another until a sample of the oil to be transferred has been collected, identified by proper labelling, and stored in a place acceptable to the Department of Environmental Protection for a minimum of fifteen days. The Board shall determine the information to be provided with each sample and may require chemical analysis of the sample.

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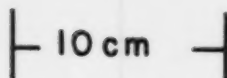
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Illustration 1. Sample Collection Device (Thief)

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TABLE 2. SAMPLING PROCEDURE

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1. Call Portland Pipe Line (PPL) and get tentative ETA (Estimated time of arrival) of tanker.
2. Check gear and equipment to ascertain all is in order.
  - a. Bottles clean and in sufficient quantity
  - b. Adequate rag supply and clean corks
  - c. Data cards and pen, shot record, etc.
  - d. Hard hat, coveralls, gloves, and flashlight (P.M.)
3. Park car in assigned area.
4. Log in with security guard and announce intent to sample.
5. Log in with dock foreman.
6. Log in with customs official if ship is not yet cleared.
7. Observing all safety rules, go aboard ship.
8. Locate chief mate or 3rd officer, and check in, identify oneself and request permission to sample the cargo.
9. Ascertain that tank to be sampled is not being emptied presently.
10. Ascertain that tank to be sampled is not under inert gass pressure. before opening hatch.
11. Open hatch and remove fire/flash screen.
12. Install bottle in sampler and attach to drop line.
13. Lower sampler into tank, until desired level is reached.
14. Actuate sampler, when bubbles stop rising, pull up sampler wiping drop line.
15. Remove bottle and wipe off, insert cork and put in box.
16. Wipe off sampler, ellage area, and any spilled oil.
17. Re-install flash-fire screen.

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18. Close hatch cover.
19. Police area sampled for rags, etc.
20. Locate chief mate and let him know you are finished.
21. Locate dock foreman and let him know you are finished.
22. Log in amount sampled and location for each tanker with dock foreman.
23. Log out at security desk.
24. Return to Laboratory and do finish copy of data form.
25. Label reference bottles (1 pint) with appropriate letter designator and date.
26. Outside of Laboratory decant sample bottles into the appropriate reference bottles.
27. Put excess oil from sample bottles into waste jar.
28. Throw oily rags into oily rag storage barrel.
29. Clean samplers and bottles with xylene 1st and hexane 2nd.
30. Replace drop line after every 4 sampling trips.
31. Log all information in ship sampling finished log, and into TRIGOM log.

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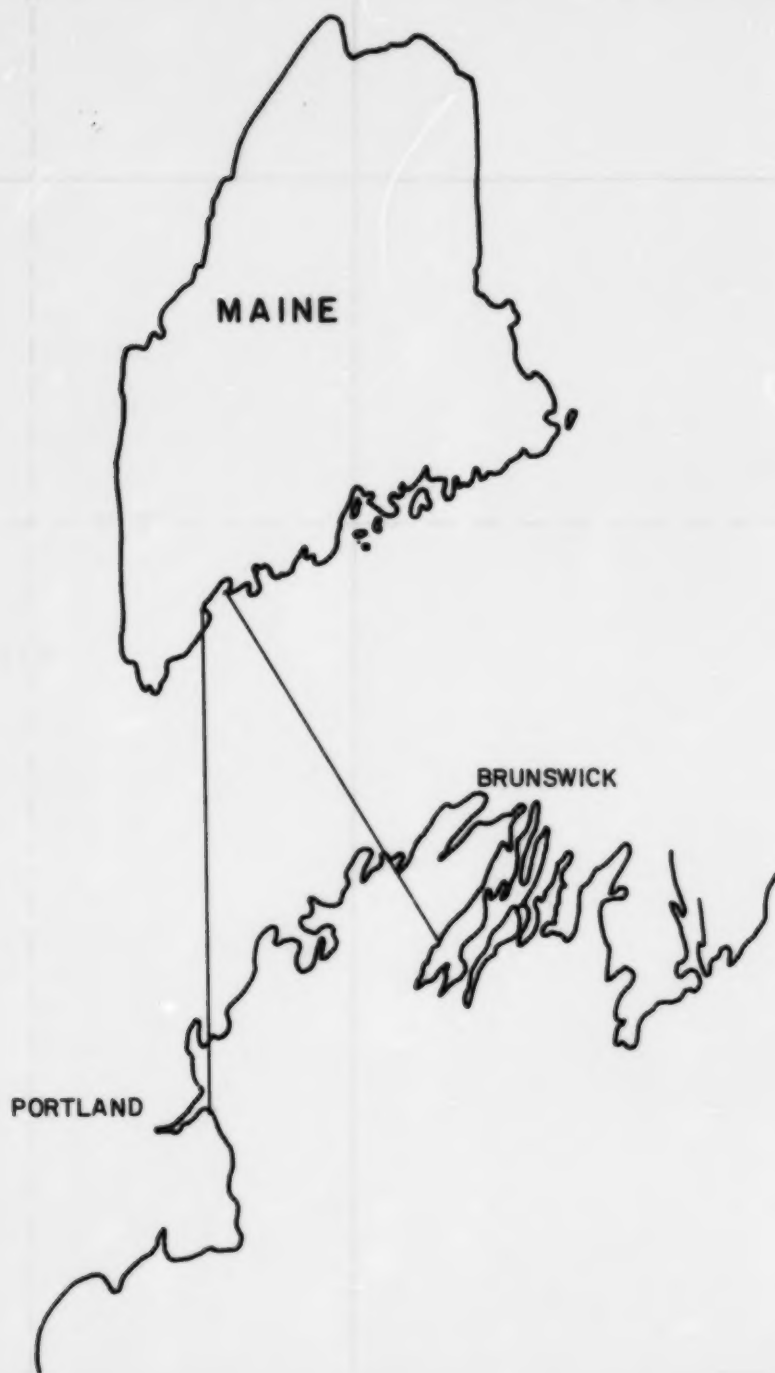
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Illustration 2. Map of Study Area

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to be of minimal risk due to the predictability and infrequency of those activities.

Floating containment was difficult. Three floating collars of 1 meter diameter corrugated aluminum culvert were constructed to contain small amounts of spilled oil. See Illustration 3. Although the collars were securely anchored on the leeward side of a massive dock, occasional heavy seas caused excessive verticle motion of the containment devices. Under those conditions any contained oil disappeared within 3 days; under less severe conditions the oil was contained longer. Attempts to reduce loss by restricting the submerged opening of the collars were unsuccessful.

Back-up spill experiments were successful. Anticipating containment problems at the South Harpswell site, back-up spill facilities were constructed at TRIGOM's South Portland laboratory. They consisted of four fiberglass tanks of 500 gallons each. A flow-through pumping station assured five exchanges per day of fresh sea-water for each tank. The tanks were located on the shore with an exposure toward the southeast.

Intertidal zone spill experiments failed. An intertidal zone spill containment device was constructed out of 0.6 meter corrugated aluminum culvert, guy wires, and cinder block anchors. See Illustration 4. The base was set in about 10 cm of gravel, resting on a clay substrate at the South Harpswell site. Although the experiment was intended to alternately expose an oil-soaked natural substrate to water and air, the containment device would not withstand any significant weather and had to be abandoned.

Important environmental data were measured. Water temperature and salinity was determined and recorded on a daily basis. Wind roses were developed from data generated by the local NOAA facilities located at the Portland International Airport. See Illustration 5 for typical example. Solar radiation was recorded during selected trials with a YSI model 31 recording pyranometer.

### SPILL PROCEDURE

Several spill series were conducted. During the course of the project 10 spills series were created at the TRIGOM location. Because of the four tanks at the facility, multiple spills were possible during each series. A total of 36 unique crudes were spilled during the 10 trials. Having abandoned the insitu floating and intertidal spill devices at South Harpswell, all spills were performed within the four tanks at TRIGOM.

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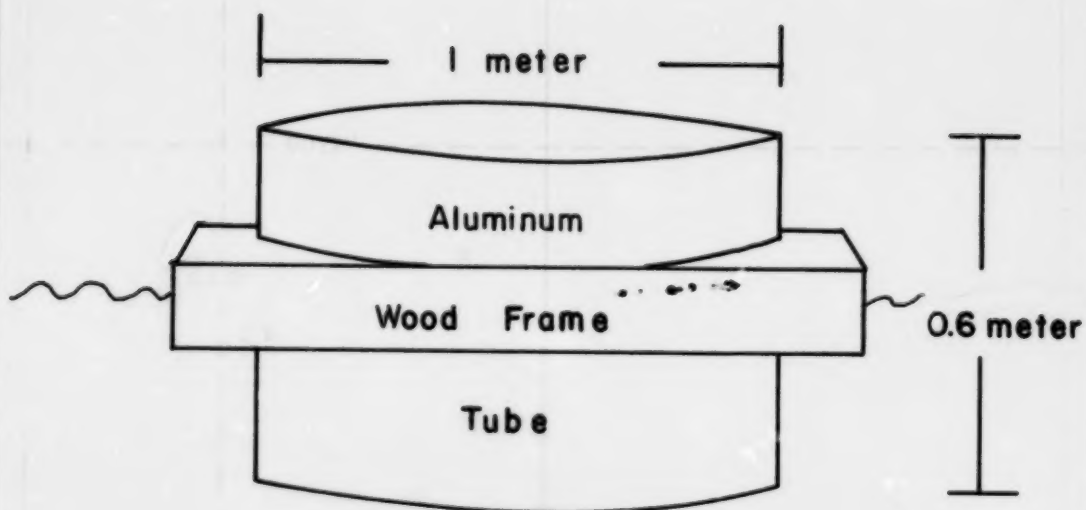
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Illustration 3. Floating Containment Device

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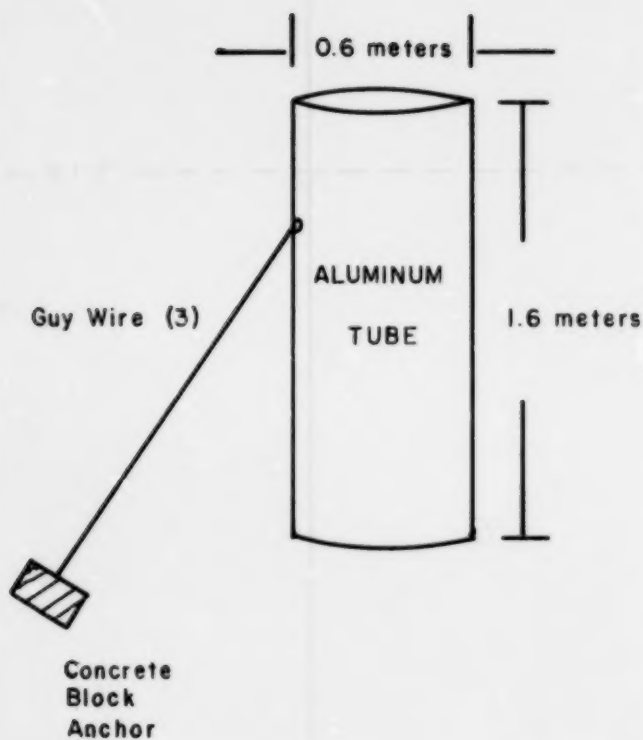
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Illustration 4. Intertidal Containment Device

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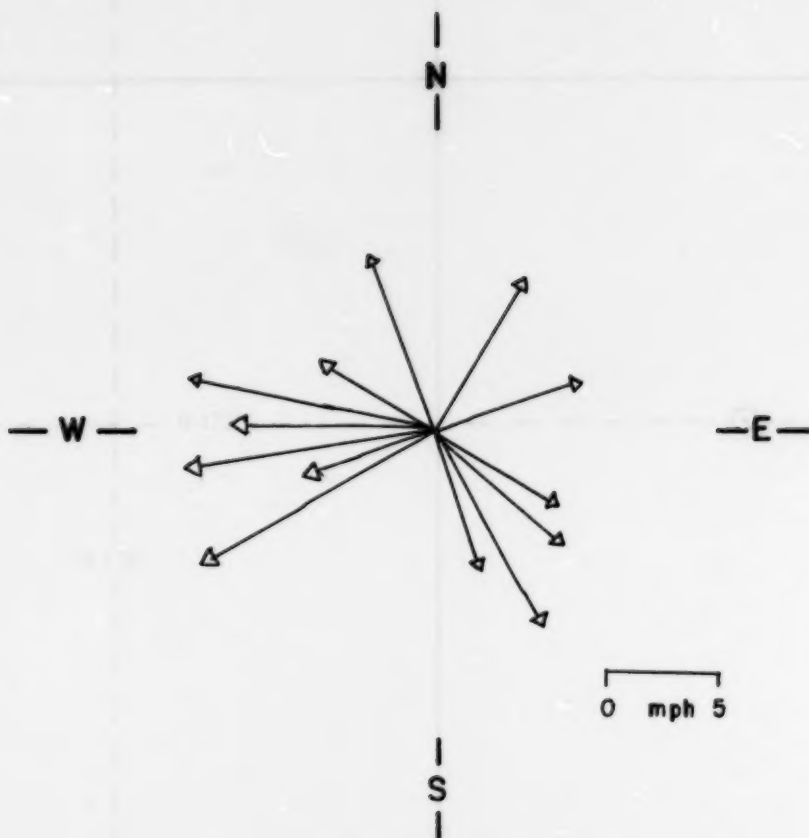
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Wind speed and direction (daily average)

| Spill age, hrs | 0    | 4    | 28   | 52   | 76   | 148  | 316  | Avg  |
|----------------|------|------|------|------|------|------|------|------|
| Salinity ‰     | 30   | 32   | 32   | 32   | 32   | 31   | 30   | 31   |
| Water T, °C    | 11.8 | 12.6 | 11.8 | 12.0 | 16.6 | 11.3 | 13.7 | 12.8 |
| Air T, °C      | 17.6 | 13.6 | 17.6 | 13.6 | 11.7 | 13.8 | 12.4 | 14.3 |

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Illustration 5. Typical Wind Rose and Environmental Data. Spill E.

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Typically, each tank contained 0.5 l of crude. Subsamples were typically taken at 0, 4, 28, 52, 76, 148 and 316 hours in pentane-washed wide mouthed glass bottles with aluminum foil lined caps. All subsamples were immediately refrigerated for temporary storage awaiting delivery for analysis. The length of each trial was limited to two weeks, coinciding with the reference holding time required by the Department of Environmental Protection regulation (see Table 1).

Spilled and reference samples were delivered to the laboratory. At the end of each spill experiments, collected subsamples and a set of reference samples, called the reference collection, were delivered to the Bowdoin laboratory for analysis. The reference collection was comprised of one sample from each tanker load of crude received at the Portland Pipe Line during a two week interval and also contained reference samples of the crudes submitted to experimental spills. The analytical task presented to the Bowdoin laboratory was to match the weathered subsamples to the proper reference sample in the reference collection.

All samples were codified. Crude samples for the reference collection were designated by the general format BBBBCCC in which BBBB is an alphanumeric combination representing the oil field origin (source) and CCC is an arbitrarily assigned tanker load number to distinguish between different loads from the same oil field. Table 3 lists all oil fields encountered during the course of the project and their codes. Spilled crudes are assigned the code format AAABBBBCCC in which AAA may be one to three digits representing the length of the spill interval in hours and BBBBCCC represents the same oil field code previously described. Table 4 shows the spill periods and the crudes.

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TABLE 3. OIL FIELD CODES

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| <u>Location</u> | <u>Code</u> | <u>Type</u>             | <u>Port of Origin</u>                      |
|-----------------|-------------|-------------------------|--------------------------------------------|
| North American  | NA01        | Transmountain Mix       | Port Moody, Vancouver, Canada              |
| South American  | SA01        | Orito                   | Tumaco, Columbia                           |
|                 | SA02        | Oriente                 | Esmeraldas, Ecuador                        |
|                 | SA03        | Guanipa                 | Puerto La Cruz, Venezuela                  |
|                 | SA04        | Ceuta                   | Puerto Miranda, "                          |
|                 | SA05        | Lagomar                 | Puerto Miranda & El Cardon, Venezuela      |
|                 | SA06        | Lagomedio               | Puerto Miranda & El Cardon, "              |
|                 | SA07        | Lagotresco              | Puerto Miranda & El Cardon, "              |
|                 | SA08        | T. J. Light             | Puerto La Salina & Amuay, Venezuela        |
|                 | SA09        | T. J. Medium            | Puerto La Salina & Amuay, "                |
|                 | SA10        | T. J. 102               | Puerto La Salina & Amuay, "                |
|                 | SA11        | Lago TI Bach            | Puerto Miranda, Venezuela                  |
|                 | SA12        | Marlago                 | Puerto Miranda, "                          |
|                 | SA13        | La Rosa Blend           | Puerta La Salina, Venezuela                |
|                 | SA14        | Mesa                    | Puerto La Cruz, Venezuela                  |
| African         | A-01        | Nigerian                | Escravos, Nigeria                          |
|                 | A-02        | Nigerian/FO             | Forcardos, Nigeria                         |
|                 | A-03        | Arzew Blend             | Arzew, Algeria                             |
|                 | A-04        | Zarzaitine              | La Skhirra, Algeria                        |
| Persian Gulf    | PG01        | Agha Jari               | Kharg Island, Iran                         |
|                 | PG02        | Light Iranian           | Kharg Island, Iran                         |
|                 | PG03        | Heavy Iranian           | Kharg Island, Iran                         |
|                 | PG12        | Iranian Blend           | Kharg Island, Iran                         |
|                 | PG04        | Basrah                  | Khor-Al-Amaja, Iraq                        |
|                 | PG05        | Light Arabian           | Ras Tanura, Saudi Arabia                   |
|                 | PG06        | Khafji                  | Ras Al Khafji (Neutral Zone), Saudi Arabia |
|                 | PG07        | Qatar                   | Umm Said, Qatar                            |
|                 | PG08        | Murban                  | Jebel Dhanna, Abu Dhabi                    |
|                 | PG09        | Zakum                   | Das Island, Abu Dhabi                      |
|                 | PG13        | Umm Shaif               | Das Island, Abu Dhabi                      |
|                 | PG10        | Dubai                   | Fateh, Dubai                               |
|                 | PG11        | Oman                    | Saih-El-Malih, Oman                        |
| Mixtures        | MX01        | Light Arabian/Lagomedio |                                            |

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TABLE 4. EXPERIMENTAL SPILLS

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| Spill<br>Series | Unknown | Weathering Times, hrs.     | Amount<br>Spilled, ml | No. Oils In<br>Reference<br>Collection |
|-----------------|---------|----------------------------|-----------------------|----------------------------------------|
| A               | SA07    | 16, 64, 370                | 470                   | 8                                      |
| B               | PG08    | 4, 64, 384                 | "                     | 15                                     |
| B               | SA04    | 1, 16, 256                 | "                     | "                                      |
| B               | (1)SA04 | 2, 8, 32                   | 940                   | "                                      |
| C               | SA05    | 72, 316                    | 470                   | 23                                     |
| C               | SA09    | 4, 28                      | "                     | "                                      |
| D               | PG02    | 0, 4, 28, 52, 76, 148, 316 | "                     | 23                                     |
| D               | PG08    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| D               | SA04    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| D               | (2)SA04 | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| D               | SA07    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| D               | (3)SA07 | ? ? ? ?                    |                       |                                        |
| E               | PG06    | 0, 4, 28, 52, 76, 148, 316 | "                     | 19                                     |
| E               | SA01    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| E               | SA06    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| E               | AO2E    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| F               | PG09    | 0, 4, 28, 52, 76, 148, 316 | "                     | 36                                     |
| F               | PG05    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| F               | SA10    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| F               | PG11    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| G               | SA05    | 0, 4, 28, 52, 76, 148, 316 | "                     | 23                                     |
| G               | SA08    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| G               | PG11    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| G               | PG09    | 0, 4, 28, 52, 76, 148, 316 | "                     | "                                      |
| O               | PG02    | 4, 28, 52, 76, 148, 316    | 940                   | 30                                     |
| O               | SA07    | 4, 28, 52, 76, 148, 316    | "                     | "                                      |
| O               | SA05    | 4, 28, 52, 76, 148, 316    | "                     | "                                      |
| O               | NA01    | 4, 28, 52, 76, 148, 316    | "                     | "                                      |

- (1) SA04 repeated at different spill concentration  
 (2) SA04 repeated same crude but different tanker load  
 (3) From an actual spill in Portland Harbor. Sample age less than 24 hrs.

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SECTION 5

PASSIVE TAGGING METHODS

DISCUSSION

Several passive tagging techniques were used. They include:

- Method 1: Numerical comparison of several n-heptadecane, n-octadecane, pristane, phytane ratios from high resolution gas chromatography.
- Method 2: Visual comparison of low and high resolution gas chromatographs and numerical comparison of V-Ni and S-N ratios.
- Method 3: Numerical comparison of V-Ni and S-N ratios and numerical comparison of high resolution gas chromatography.
- Method 4: Visual comparison of infrared spectra.

Techniques were chosen according to their availability, simplicity, and adaptability to the often limited technical resources of a regulatory agency such as Maine's Department of Environmental Protection.

Gas chromatography is an important tagging method. Blumer has pointed out that gas chromatographic analysis theoretically is ideally suited for application to spill-source correlations. In principle, gas chromatography allows for rapid straightforward, concentration determination of numerous components of petroleum oils. The use of relative concentration values for pattern recognition (finger printing) depends on the fact that only those oil samples with identical chemical composition can have had identical geochemical histories. Therefore, fingerprints based on component concentration values very likely will be unique for a specific geographic location or field. Of paramount concern is the ability of those compounds in the fingerprinting region of the chromatogram to withstand environmental loss or degradation (weathering). The procedure developed by Zafiriou takes advantage of the relative concentration determination of the complex mixture of compounds present in the intermediate molecular weight fraction. Most of these compounds are stable, mutually soluble and relatively water-insoluble substances. Furthermore, the volatility of

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these substances is such that the analysis by high resolution gas chromatography can be carried out in an accurate and reproducible fashion. As proposed, the normal hydrocarbons C<sub>17</sub> and C<sub>18</sub> (n-heptadacane and n-octadacane) and two isoprenoid hydrocarbons pristane (C<sub>19</sub>) and phytane (C<sub>20</sub>) were the primary fingerprinting agents. The choice of these compounds was dictated by the following requirements: first, the compounds should occur in the very large majority of all petroleum oils; second, they should be found in relatively large concentrations; third, rather substantial changes in relative concentrations should be expected to occur between different oil fields; fourth, as mentioned above these compounds should be stable, insoluble in water and non-volatile; fifth, they should elute during gas chromatographic analysis over a relatively short time span to insure base-line stability and increase the accuracy of the concentration determination; and sixth, they should be capable of good resolution from any contaminating components and from each other. The proposed set of four compounds appear to be the optimum choice available at the present state of the art in gas chromatography.

Chromatographic finger printing has been tested. The method was initially evaluated by a joint project between the USEPA (Edison, N.J.) and Zafiriou (1). A variety of commonly encountered oils and oil products were exposed to weathering and submitted to passive tagging by gas chromatography. In comparing 35 weathered samples to 16 reference samples, 74% success was achieved. Additional studies were conducted by Zafiriou testing the technique on 22 unweathered, samples of crude oil. Because several of the samples were replicates from identical sources the tagging effort was only 25% successful and seemed to lack resolution power between very similar samples. Nevertheless the technique appeared to have good potential as a discriminatory tool that might be developed to meet the needs of the Maine situation. Consequently, the tagging procedure proposed by Zafiriou became the object of intensive investigation and evaluation during the course of this study.

Other passive tagging techniques were available. Initial studies by Mayo indicated that a very characteristic collection of weak absorption bands with fingerprinting potential occurs in the 600-400 cm<sup>-1</sup> region of the infrared spectrum of crude oils (2). Absorption in this region was shown to be due to stable aromatics which, if able to withstand weathering, might become useful passive tagging tool. Ni-V and S-N ratios have long been proposed for passive tagging. Studies by Lieberman showed limited success (3). Despite the lack of promise as an especially powerful tool, nevertheless it was felt that the method warranted further investigation to determine its possible role as a supplemental, supportive technique.

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**EXPERIMENTAL**

**Method 1**

Zafirion proposed OV-101 SCOT chromatographic columns (4). This approach had been shown to produce adequate C-17, C-18, pristane and phytane resolution. However, because of the high operating costs of SCOT columns, a re-evaluation of the conditions developed by Blumer for the use of Apiezon-L as a stationary phase on packed columns was explored. It was found that a significant improvement in column resolution is achieved by applying the stationary phase to the support material by filtration techniques (5). Indeed, by employing a 12 foot x 1/8 inch column packed with 3% Apiezon-L on chromasorb-W it has been possible to achieve resolution of the pristane-C<sub>17</sub> and phytane-C<sub>18</sub> peaks previously only attained with SCOT or capillary column techniques. See Illustration 6. Similar resolving power was also achieved by employing OV-1 as the stationary phase on a 6 foot column. This initial success at improved resolution of the four key peaks led to a considerable amount of work employing matched column techniques in an attempt to substitute packed for SCOT columns. Unfortunately, considerable variation in resolution was found between columns which, as they were being used at their maximum efficiency, limited intercolumn comparisons. Furthermore, column durability which was hoped to be improved over SCOT type columns did not, in fact, appear to be as good.

Inorganic eutectic phases were evaluated. It has been noted by Zafirion that polar stationary phases (such as FFAP) can separate the pristane C<sub>17</sub> and phytane-C<sub>18</sub> peaks with better resolution than the non-polar phases, but that temperature instability and bleed characteristics are unfavorable. Columns employing inorganic eutectic mixtures of NiNO<sub>3</sub>, LiNO<sub>3</sub>, and KNO<sub>3</sub>, at a 18:27:55 ratio as a stationary phase were shown by Koons and others to have very high resolving power and could completely separate the four components of interest (6). As these systems were expected to also possess high thermal stability and good bleed characteristics, the ideal packed column appeared to be a possibility. Although the initial results with synthetic mixtures seemed extremely promising, it was found when dealing with complex petroleum hydrocarbons that "pump" effects (shifts in retention time) were observed, particularly in the chromatographic regions of interest. All attempts to avoid these chromatographic distortions failed and the use of inorganic eutectics was abandoned. See Illustration 7 for a typical chromatogram.

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3. PHYTANE
4. C18

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Illustration 6. Chromatogram of Orito Crude with 4 meter Apiezon L Packed Column.

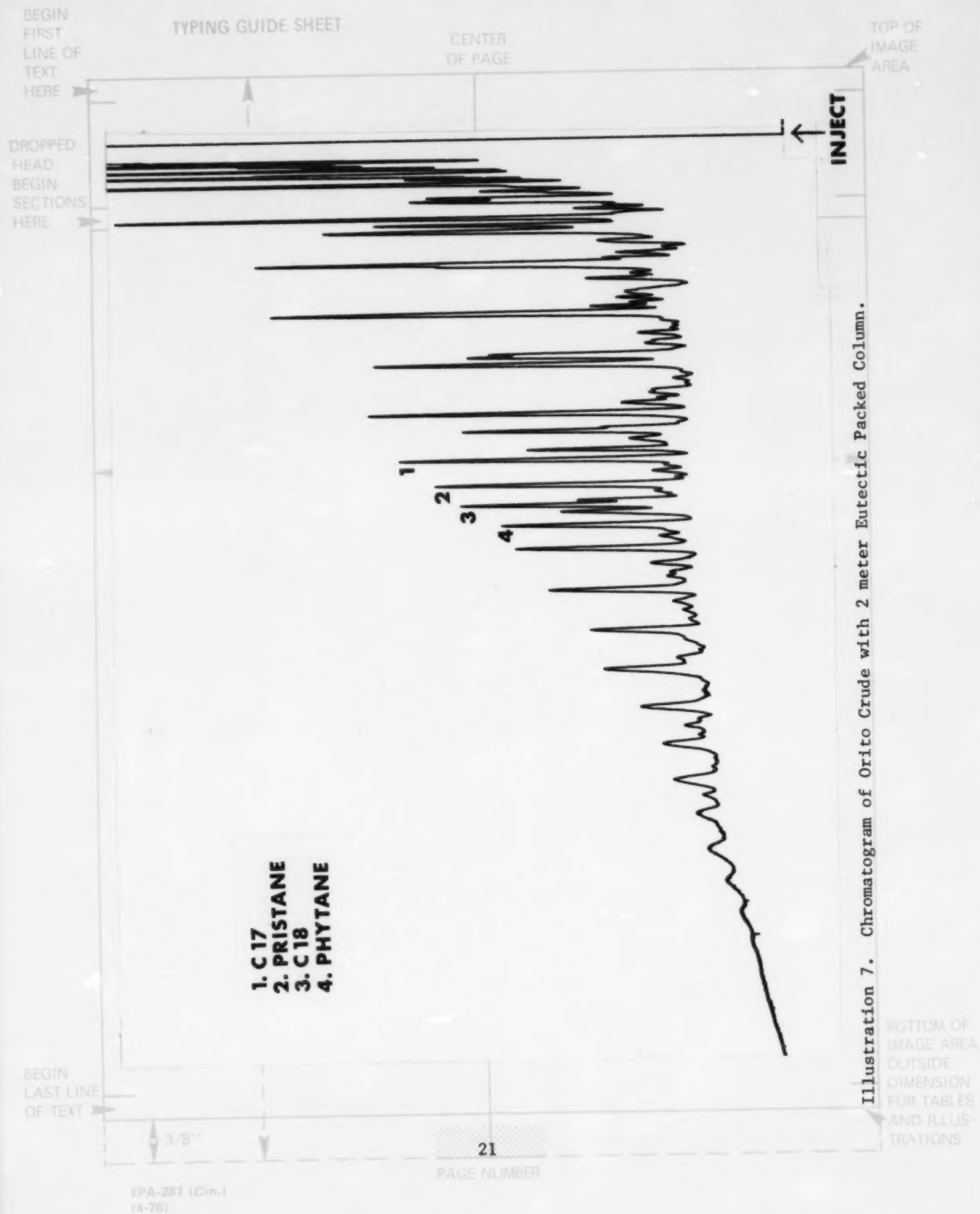


Illustration 7. Chromatogram of Orito Crude with 2 meter Eutectic Packed Column.

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SCOT OV-1 columns were chosen. After showing that no available packed column was capable of the necessary resolution or dependability, the SCOT system became necessary. The studies of Zafiriou had been performed with an OV-101 stationary phase SCOT system. However, after extensive evaluations the OV-1 stationary phase SCOT column appeared somewhat superior in stability, resolution, and bleed characteristic while retaining the desirable chromatographic characteristics of the OV-101. Therefore, the SCOT-OV-1 system became the primary working column for high resolution chromatography evaluation.

Slightly deviating from Zafiriou, oil samples were taken up in and diluted with carbon disulfide. Any remaining water was decanted and the disulfide layer dried over anhydrous sodium sulfate. Sulfur was removed by sample elution through an activated copper column with carbon disulfide. Finally, samples were directly injected into a Perkin Elmer model 900 or model 990 gas chromatograph with SCOT OV-1 capillary columns. The high resolution gas chromatograms were evaluated and compared by a numerical technique to be discussed in detail later. The relative concentrations of C-17, pristane, C-18, and phytane were determined from peak heights of the four components above a base line determined from a blank-run chromatogram. Comparisons between chromatograms were made based on the calculated ratios C-17/Pristane, Pristane/Phytane, Phytane/C-18, and C-17/C-18 thereby eliminating the need for precise attenuation control. See Illustration 8.

Method 2

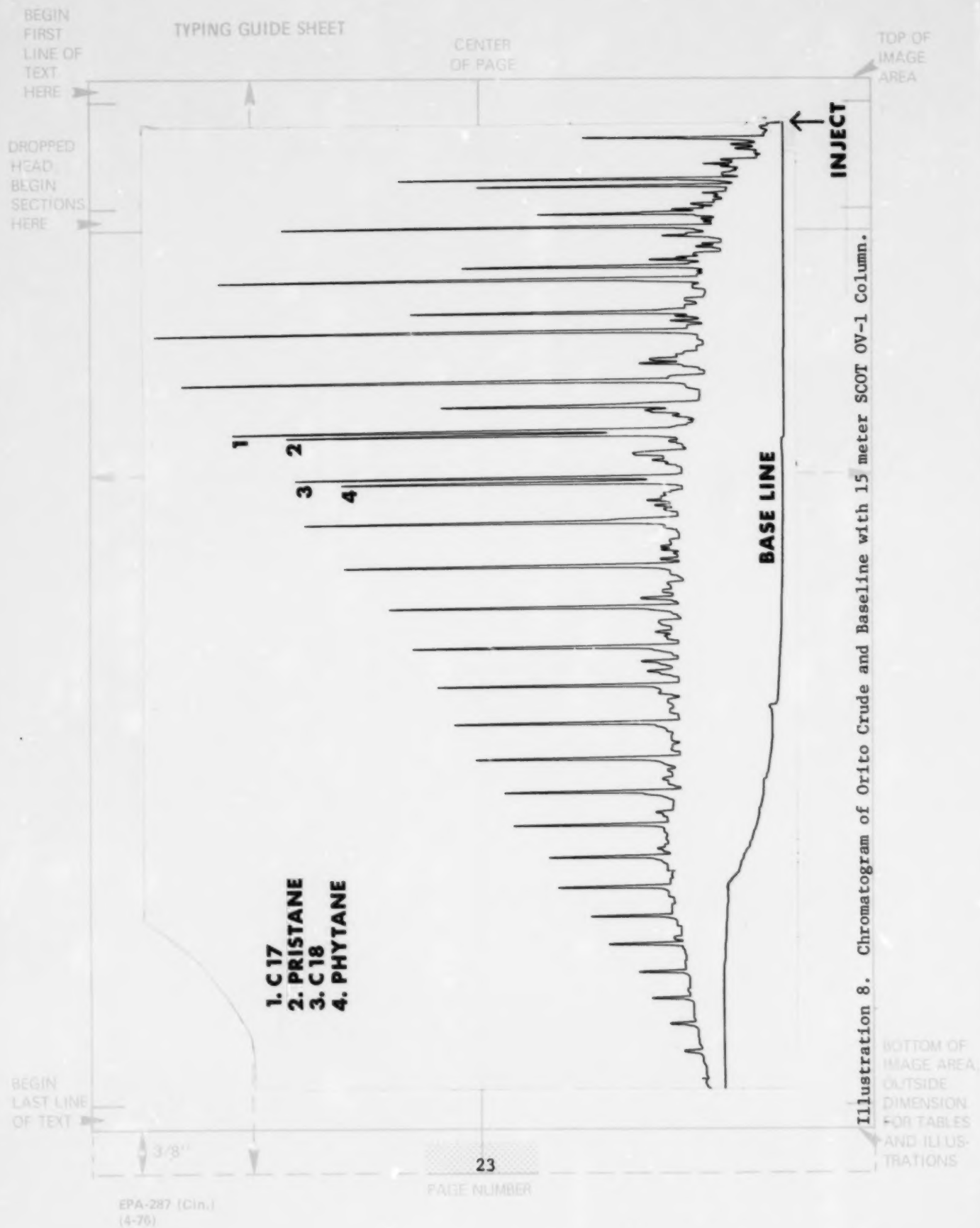
The oil-water mixture was extracted with chloroform then centrifuged, filtered, and stripped at 280°C with nitrogen. Alternatively, carbon disulfide was used in place of chloroform. Again, sulfur was removed by sample elution through an activated copper column with carbon disulfide. The stripped sample was submitted to an analytical routine of comparing unknown oils to a reference collection, first, by visual inspection of low resolution chromatograms, then second, by visual inspection of high resolution chromatograms (7), and third, by numerical comparisons of V-Ni and S-N ratios (8).

More specifically, both unknown and reference samples were submitted first to packed column chromatography. The stationary phase was established as 10% OV-101 on Chromosorb W. An overall visual comparison of the chromatograms plus comparison of retention times of major peaks were used to match the unknown with similar crude types. Based upon the comparisons, the unknown is reported to be either (a) unlike the reference sample or (b) like the reference sample in enough respects to be considered for further examination. Following the initial sorting process, reference samples which were selected as matching candidates as well as the unknown were subject to high resolution gas chromatography. All samples were first column chromatographed

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on silica gel/alumina to remove the aromatic fraction of the petroleum residue and then gas chromatographed on SCOT OV-101 column as in Method 1. To eliminate operator variance, the Perkin Elmer Model MS-41 solid sampler was used. The comparison was again based on a visual assessment of differences between the chromatograms of the unknown and reference samples. Samples of unknown origin were reported to be either (a) unlike or (b) like a reference sample or samples.

The final screening was based upon similarities between N-Ni and S-N ratios. For the nickel and vanadium determinations, the samples were ashed in the presence of benzene sulfonic acid followed by additional heating to burn off carbon leaving an inorganic residue. The residue was dissolved in hydrochloric acid and the nickel-vanadium concentrations determined by a Perkin Elmer Model 601 atomic absorption spectrophotometer. To determine the sulfur and nitrogen values, the samples were analyzed by routine bomb-SO<sub>2</sub> and Kjeldahl-nitrogen microanalytical techniques at Schwarzkopf Microanalytical Laboratory, Woodside, N. Y., 11377.

#### Method 3

This method combined Method 1 (numerically evaluated high resolution gas chromatography) with the V-Ni and S-N ratio portion of Method 2.

#### Method 4

Infrared fingerprints were evaluated in the 600-400 cm region. The infrared absorption spectra were determined on weathered oil samples which were prepared by the same procedure used for gas chromatography in Method 2 except that samples were not column chromatographed to remove aromatics. Solvent was removed prior to spectral studies by stripping with nitrogen under reduced pressure. Spectra were obtained in potassium bromide cells employing scale expansion techniques on a Perkin Elmer model 621 infrared spectrophotometer. See typical infrared spectra in Illustration 9.

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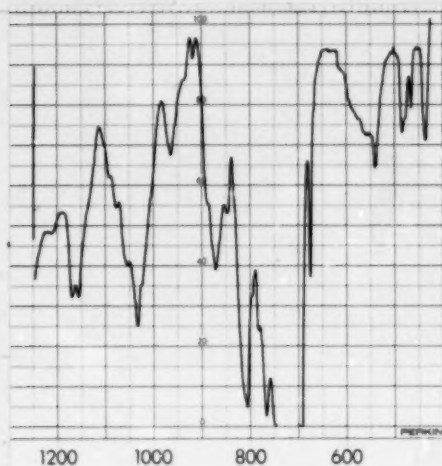
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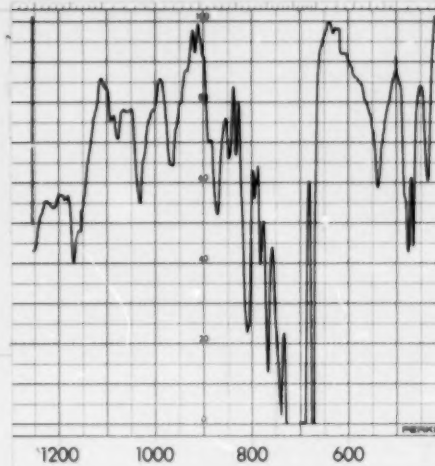
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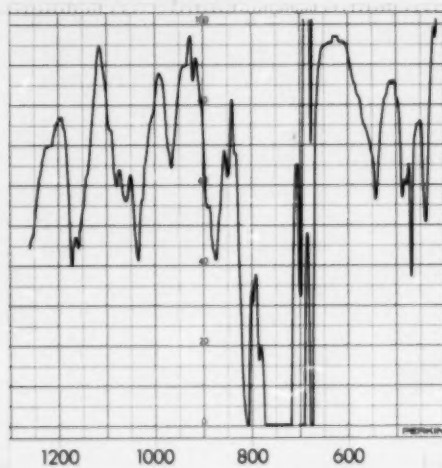
**BASRAH CRUDE**



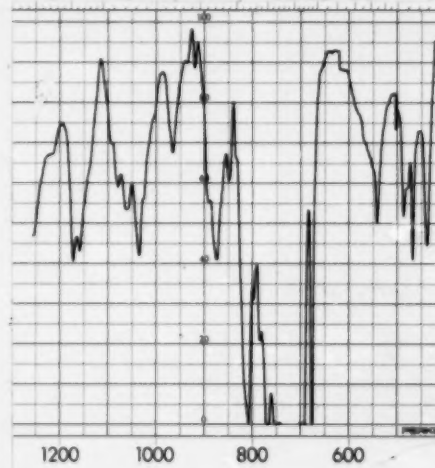
**NIGERIAN CRUDE**



**ZAKUM CRUDE**



**QATAR CRUDE**



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**Illustration 9. Infrared Spectra of Several Crudes.**

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SECTION 6

PASSIVE TAGGING METHODS EVALUATION

METHOD 1: NUMERICAL EVALUATION OF HIGH RESOLUTION CHROMATOGRAPHY.

Two preliminary spills of Venezuelan crude oils, Lagomar and Cueta, were evaluated. In addition to some apparent deterioration of C-17/Pristane, pristane/phytane, phytane/C18 and C-17/C-18 ratios, during weathering (discussed later) difficulties were encountered when attempting to compare ratios from the unknown to a large collection of reference ratios. See Table 5. The problem of objectively evaluating the degree of match between ratios visually, particularly in the case of a close match, dictated the need for a more rigorous, mathematical treatment. Consequently, an approach was developed which requires calculating the ratios (C-17/pristane, pristane/phytane, phytane/C-18 and C-17/C-18) for both the unknown sample and the reference samples and determining the ratio of the corresponding ratios. The deviation of the ratio of the ratios from unity is determined for each point, squared, and summed to establish a "residual" value. Mathematically,

$$R = \sum_{i=1}^n \left(1 - \frac{r_u}{r_k}\right)^2$$

where R = residual value

i = the ratio C-17/pristane, pristane/phytane, phytane/C-18 or C-17/C-18.

$r_u$  = the value of the ith ratio for an unknown sample,

$r_k$  = the value of the ith ratio for the known (reference) sample.

The larger the magnitude of the residual, the poorer the match of the unknown with the known reference sample. Conversely, the smaller the residual, the better the match. For a perfect match  $R = 0$ . In this manner a reference collection can be compared and ranked against an unknown. This method is especially well suited to comparing an unknown to sets of reference data and, subsequently, computer analysis. Typically, the reference collection is ranked according to increasing values of R. Percent deviations from the smallest value of R may be calculated to facilitate comparison. See Table 6 for a presentation of data from Spill A.

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TABLE 5. LIST OF RATIOS FROM SPILL A

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Average Ratio, N = 8 to 11

| <u>Spilled<br/>Crude<br/>Type</u> | <u>Spill<br/>Location</u> | <u>PR<br/>PH</u> | <u>C17<br/>PR</u> | <u>C18<br/>PH</u> | <u>C17<br/>C18</u> |
|-----------------------------------|---------------------------|------------------|-------------------|-------------------|--------------------|
| Nigerian F/O                      | So. Portland              | --               | .88               | --                | .95                |
|                                   | So. Harpswell             | --               | .90               | --                | .97                |
| Lagomar                           | So. Portland              | --               | 1.89              | --                | .95                |
|                                   | So. Harpswell             | --               | 1.85              | --                | .96                |
| Lagomar                           | So. Portland              | .92              | 1.43              | 1.22              | 1.08               |
| Ceuta                             | So. Portland              | .96              | 1.34              | 1.25              | 1.03               |
| <u>Reference<br/>Crude Type</u>   |                           |                  |                   |                   |                    |
| Qatar                             |                           | 1.02             | 3.43              | 2.67              | 1.31               |
| Ceuta                             |                           | 1.02             | 1.91              | 1.67              | 1.17               |
| Lagomar                           |                           | 0.96             | 2.04              | 1.57              | 1.25               |
| Lagomedio                         |                           | 0.94             | 1.47              | 1.23              | 1.12               |
| T. J. 102                         |                           | 0.89             | 1.50              | 1.10              | 1.21               |
| Lagotreco, load #30               |                           | 1.13             | 1.38              | 1.42              | 1.09               |
| Lagotreco, load #53               |                           | 1.17             | 1.64              | 1.73              | 1.11               |
| Lagotreco, load #56               |                           | 1.16             | 1.79              | 1.82              | 1.13               |

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TABLE 6. R VALUES, SPILL A METHOD 1A

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Type  
of Oil

CODE

Number of  
replicates

(1)  
R

Qatar

PG07

1

0.59375

Ceuta

SA04

1

0.10522

Lagomar

SA05

1

0.14390

Lagomedio

SA06

1

0.07273

Lagotreco

SA07

3

(0.03775)

T. J. 102

SA10

1

0.15585

(1) 3 unknown replicates

\_\_\_\_\_ = correct match

( ) = lowest residual

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Method 1A

Although several methods of data treatment are possible, two of the most successful are described here. In the simplest approach, peak height values from chromatograms replicate samples from the unknown spill were arithmetically averaged and used to calculate the average values of  $r_u$ . Mathematically the difference between  $r_u$  calculated from averaged peak heights and an average  $r_u$  calculated from individual values of  $r_u$  is trivial. Similarly, average values of  $r_k$  for the reference collection were determined for each unique oil field using chromatograms from replicate samples. The assumption was also made that replicate loads from the same oil field could properly be used to determine the best estimate of  $r_k$  for that source. Tables 6,7,8,9, and 10 show the results of this approach to predict a match in spill series A,B,C,D, and E respectively. For example in spill series C, Table 8, two unknowns were satisfactorily matched. Clearly, the match was very positive, the correct residual value being significantly smaller than the nearest mismatch. In all, 14 out of 16 matching exercises were successful for an 88 percent score. Results are summarized in Table 11. Clearly, this method does not permit an attempt to resolve different tanker loads from the same oil field.

Method 1B

A second method of data treatment is highly discriminating. It was observed that variations in R between different tanker loads from the same oil field often exceeded variations in R between different oil fields. However, it may be possible to distinguish not only the type of oil spilled (oil field) but also to distinguish the correct tanker load of several from the same oil field. First, N replicate samples of the spill are compared to the reference collection, thus generating N sets of residuals as shown in Table 12. In this example, replicates of the unknown were selected from the weathered unknown collection, the assumption being made that weathering would not adversely affect results. The affect of weathering will be described later. The best measurement of match between an unknown and oil types (field) is represented by the average value of minimum R values from N sets without regard to tanker number. Typically, referring to Table 12, the best match between the unknown and SA07 (Lagotrec) is

$$R = \frac{0.019963 + 0.030508 + \dots 0.043663}{7} = 0.030962$$

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TABLE 7. R VALUES, SPILL B METHOD 1A

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| Type<br>of Oil | CODE | Number of<br>replicates | (1)<br>R  | (2)<br>R  | (3)<br>R  |
|----------------|------|-------------------------|-----------|-----------|-----------|
| Nigerian       | A-01 | 1                       | 1.5709    | 0.34903   | 0.34296   |
| Nigerian/FO    | A-02 | 1                       | 4.2399    | 0.63744   | 0.63420   |
| Murban         | PG08 | 2                       | (0.06649) | 0.20478   | 0.20408   |
| Dubai          | PG10 | 1                       | 0.91692   | 0.00686   | 0.00653   |
| Guanipa        | SA03 | 3                       | 2.8278    | 0.45937   | 0.45685   |
| Ceuta          | SA04 | 3                       | 1.2373    | 0.00365   | (0.00248) |
| Lagomar        | SA05 | 2                       | 1.0441    | 0.01748   | 0.01844   |
| Lagotreco      | SA07 | 1                       | 1.1810    | (0.00325) | 0.00286   |
| T. J. Light    | SA08 | 1                       | 0.85181   | 0.00969   | 0.00803   |

(1) 3 unknown replicates

\_\_\_\_\_ = correct match

( ) = lowest residual

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TABLE 8. R VALUES, SPILL C METHOD 1A

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| Type<br>of Oil  | CODE | Number of<br>replicates | (1)<br>R  | (2)<br>R  |
|-----------------|------|-------------------------|-----------|-----------|
| Nigerian        | A-01 | 1                       | 0.40322   | 0.34929   |
| Lt. Ar./Lagomed | MX01 | 1                       | 0.09409   | 0.02489   |
| Lt. Iranian     | PG02 | 1                       | 0.04116   | 0.01660   |
| Murban          | PG08 | 2                       | 0.35971   | 0.23727   |
| Zakum           | PG09 | 1                       | 0.39313   | 0.26835   |
| Oman            | PG11 | 1                       | 0.09632   | 0.02666   |
| Orito           | SA01 | 1                       | 0.09382   | 0.21717   |
| Guanipa         | SA03 | 1                       | 0.41430   | 0.46231   |
| Ceuta           | SA04 | 3                       | 0.06629   | 0.00956   |
| Lagomar         | SA05 | 2                       | 0.03766   | (0.00674) |
| Lagomedio       | SA06 | 1                       | 0.10066   | 0.03340   |
| Lagotreco       | SA07 | 3                       | 0.10534   | 0.03449   |
| T. J. Light     | SA08 | 2                       | 0.07711   | 0.01454   |
| T. J. Medium    | SA09 | 1                       | (0.00688) | 0.03952   |
| Lago T.I. Bach  | SA11 | 1                       | 0.09305   | 0.03255   |

(1) 3 unknown replicates

(2) 2 unknown replicates

\_\_\_\_\_ = correct match

( ) = lowest residual



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TABLE 10. R VALUES, SPILL E METHOD 1A

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| Type<br>of Oil | CODE | Number of<br>Replicates | (1)<br>R  | (1)<br>R  | (1)<br>R  | (1)<br>R  |
|----------------|------|-------------------------|-----------|-----------|-----------|-----------|
| Nigerian       | A-01 | 2                       | 2.5803    | 0.18208   | 0.60177   | 0.07115   |
| Nigerian/FO    | A-02 | 1                       | 5.6477    | 0.26815   | 1.5435    | (0.00297) |
| Khafji         | PG06 | 1                       | (0.01831) | 0.57550   | 0.13434   | 1.7591    |
| Oman           | PG11 | 1                       | 1.0990    | 0.08864   | 0.007856  | 0.7011    |
| Orito          | SA01 | 1                       | 2.7787    | (0.01962) | 0.51312   | 0.27682   |
| Guanipa        | SA03 | 1                       | 4.0164    | 0.22988   | 1.0491    | 0.02020   |
| Ceuta          | SA04 | 3                       | 1.3035    | 0.06984   | 0.11803   | 0.72470   |
| Lagomar        | SA05 | 3                       | 1.2465    | 0.09543   | 0.10363   | 0.97164   |
| Lagomedio      | SA06 | 1                       | 0.61806   | 0.20574   | (0.01258) | 1.1697    |
| Lagotresco     | SA07 | 3                       | 1.4704    | 0.05138   | 0.16381   | 0.49594   |
| T.J. Light     | SA08 | 1                       | 1.1880    | 0.07960   | 0.09445   | 0.71881   |
| T.J. 102       | SA10 | 1                       | 3.8809    | 0.06112   | 0.82425   | 0.91700   |

(1) 7 unknown replicates

\_\_\_\_\_ = correct match

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TABLE 11. SCORE SUMMARY METHOD 1A

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| <u>Trial<br/>(Spill)</u> | <u>No. in<br/>Reference<br/>Collection</u> | <u>Final<br/>Choice</u> | <u>Correct<br/>Match</u> |
|--------------------------|--------------------------------------------|-------------------------|--------------------------|
| 1                        | 6                                          | SA07                    | SA07                     |
| 2                        | 9                                          | PG08                    | PG08                     |
| 3                        | 9                                          | SA07                    | SA04                     |
| 4                        | 9                                          | SA04                    | SA04                     |
| 5                        | 15                                         | SA09                    | SA09                     |
| 6                        | 15                                         | SA05                    | SA05                     |
| 7                        | 15                                         | PG02                    | PG02                     |
| 8                        | 15                                         | PG08                    | PG08                     |
| 9                        | 15                                         | SA04                    | SA04                     |
| 10                       | 15                                         | SA04                    | SA04                     |
| 11                       | 15                                         | SA11                    | SA07                     |
| 12                       | 15                                         | SA07                    | SA07                     |
| 13                       | 12                                         | PG06                    | PG06                     |
| 14                       | 12                                         | SA01                    | SA01                     |
| 15                       | 12                                         | SA06                    | SA06                     |
| 16                       | 12                                         | A-02                    | A-02                     |

Score = 14/16 correct = 88 percent

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TABLE 12. R VALUES, SPILL D METHOD 1B

| 0 HR. Unknown | 4 HR. Unknown | 28 HR. Unknown | 52 HR. Unknown |
|---------------|---------------|----------------|----------------|
| CODE          | CODE          | CODE           | CODE           |
| 1 SA04120     | 1 SA04133     | 1 SA04120      | 1 SA04133      |
| 2 SA09112     | 2 SA04120     | 2 SA04133      | 2 SA04120      |
| 3 SA04115     | 3 SA04115     | 3 SA09112      | 3 SA04115      |
| 4 SA04133     | 4 MX01119     | 4 SA04115      | 4 MX01119      |
| 5 SA07118     | 5 PG02113     | 5 SA07118      | 5 SA05111      |
| 6 SA11114     | 6 SA05111     | 6 PG02113      | 6 PG02113      |
| 7 MX01119     | 7 SA04125     | 7 MX01119      | 7 PG11116      |
| 8 SA05111     | 8 SA08121     | 8 SA05111      | 8 SA08121      |
| 9 PG02113     | 9 PG11116     | 9 SA04125      | 9 SA07118      |
| 10 PG11116    | 10 SA07118    | 10 SA11114     | 10 SA04125     |
| 11 SA08121    | 11 SA11114    | 11 SA08121     | 11 SA11114     |
| 12 SA04125    | 12 SA05127    | 12 PG11116     | 12 SA09112     |
| 13 SA08126    | 13 SA09112    | 13 SA05127     | 13 SA05127     |
| 14 SA07132    | 14 SA08126    | 14 SA08126     | 14 SA06130     |
| 15 SA06130    | 15 SA06130    | 15 SA06130     | 15 SA08126     |
| 16 SA05127    | 16 SA07132    | 16 SA01123     | 16 SA07132     |
| 17 SA07128    | 17 SA07128    | 17 SA07132     | 17 SA07128     |
| 18 SA01123    | 18 SA01123    | 18 SA07128     | 18 SA01123     |
| 19 PG08131    | 19 PG08131    | 19 PG08131     | 19 PG08131     |
| 20 PG08117    | 20 PG08117    | 20 PG08117     | 20 PG08117     |
| 21 A-01122    | 21 PG09124    | 21 A-01122     | 21 PG09124     |
| 22 PG09124    | 22 A-01122    | 22 PG09124     | 22 A-01122     |
| 23 SA03129    | 23 SA03129    | 23 SA03129     | 23 SA03129     |
| R             | R             | R              | R              |
| 0.004073      | 0.000969      | 0.009124       | 0.000195       |
| 0.010114      | 0.007854      | 0.016289       | 0.007632       |
| 0.010181      | 0.013208      | 0.024593       | 0.011072       |
| 0.011785      | 0.015574      | 0.034172       | 0.015428       |
| 0.019963      | 0.019508      | 0.039255       | 0.015742       |
| 0.032597      | 0.019698      | 0.041425       | 0.017668       |
| 0.043080      | 0.025824      | 0.056451       | 0.028826       |
| 0.043932      | 0.029016      | 0.057557       | 0.029551       |
| 0.052548      | 0.030072      | 0.065972       | 0.032663       |
| 0.053658      | 0.030508      | 0.069576       | 0.033775       |
| 0.055914      | 0.038438      | 0.078520       | 0.036811       |
| 0.060334      | 0.045504      | 0.080020       | 0.042744       |
| 0.092125      | 0.046990      | 0.094805       | 0.052763       |
| 0.093214      | 0.055060      | 0.109333       | 0.056188       |
| 0.097069      | 0.055064      | 0.114195       | 0.062018       |
| 0.102466      | 0.087500      | 0.134357       | 0.091586       |
| 0.115905      | 0.097758      | 0.137561       | 0.101341       |
| 0.121346      | 0.230231      | 0.156715       | 0.222238       |
| 0.364047      | 0.286144      | 0.369210       | 0.294537       |
| 0.436744      | 0.378757      | 0.472380       | 0.382214       |
| 0.452511      | 0.498963      | 0.499946       | 0.506507       |
| 0.567122      | 0.509404      | 0.588947       | 0.516582       |
| 0.672409      | 0.843415      | 0.680975       | 0.857527       |

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316 HR. Unknown

|    | CODE    | R        |
|----|---------|----------|
| 1  | SA04133 | 0.008478 |
| 2  | SA04120 | 0.012293 |
| 3  | PG02113 | 0.021482 |
| 4  | SA04115 | 0.030186 |
| 5  | MX01119 | 0.033492 |
| 6  | SA05111 | 0.035842 |
| 7  | SA04125 | 0.042588 |
| 8  | SA07118 | 0.043663 |
| 9  | SA09112 | 0.048520 |
| 10 | SA08121 | 0.053315 |
| 11 | PG11116 | 0.055586 |
| 12 | SA05127 | 0.058562 |
| 13 | SA11114 | 0.063691 |
| 14 | SA06130 | 0.079592 |
| 15 | SA08126 | 0.080114 |
| 16 | SA07132 | 0.123321 |
| 17 | SA07128 | 0.133158 |
| 18 | SA01123 | 0.212035 |
| 19 | PG08131 | 0.313975 |
| 20 | PG08117 | 0.421212 |
| 21 | A-01122 | 0.531031 |
| 22 | PG09124 | 0.535970 |
| 23 | SA03129 | 0.813006 |

148 HR. Unknown

|    | CODE    | R        |
|----|---------|----------|
| 1  | SA04120 | 0.002998 |
| 2  | SA04133 | 0.004627 |
| 3  | SA04115 | 0.017990 |
| 4  | SA09112 | 0.027175 |
| 5  | SA07118 | 0.028230 |
| 6  | PG02113 | 0.029119 |
| 7  | MX01119 | 0.033437 |
| 8  | SA05111 | 0.036020 |
| 9  | SA04125 | 0.043583 |
| 10 | SA11114 | 0.047713 |
| 11 | SA08121 | 0.050890 |
| 12 | PG11116 | 0.051892 |
| 13 | SA05127 | 0.070448 |
| 14 | SA08126 | 0.079993 |
| 15 | SA06130 | 0.083371 |
| 16 | SA07132 | 0.107905 |
| 17 | SA07128 | 0.122916 |
| 18 | SA01123 | 0.167144 |
| 19 | PG08131 | 0.330069 |
| 20 | PG08117 | 0.425834 |
| 21 | A-01122 | 0.492848 |
| 22 | PG09124 | 0.545438 |
| 23 | SA03129 | 0.739970 |

76 HR. Unknown

|    | CODE    | R        |
|----|---------|----------|
| 1  | SA04133 | 0.002140 |
| 2  | SA04115 | 0.004811 |
| 3  | SA04120 | 0.008439 |
| 4  | MX01119 | 0.011793 |
| 5  | SA05111 | 0.017643 |
| 6  | PG11116 | 0.019999 |
| 7  | SA08121 | 0.020045 |
| 8  | SA04125 | 0.022134 |
| 9  | SA07118 | 0.022453 |
| 10 | SA11114 | 0.022979 |
| 11 | PG02113 | 0.029420 |
| 12 | SA09112 | 0.044513 |
| 13 | SA08126 | 0.045761 |
| 14 | SA06130 | 0.048654 |
| 15 | SA05127 | 0.049124 |
| 16 | SA07132 | 0.068729 |
| 17 | SA07128 | 0.077721 |
| 18 | SA01123 | 0.230013 |
| 19 | PG08131 | 0.282161 |
| 20 | PG08117 | 0.361060 |
| 21 | A-01122 | 0.483941 |
| 22 | PG09124 | 0.486337 |
| 23 | SA03129 | 0.834143 |

NOTE: One chromatogram for all  
unknown and reference  
samples

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Likewise for SA04 (Cueta),

$$R = \frac{0.004073 + 0.000969 + \dots 0.008478}{7} = 0.003997$$

In this method, the candidate crudes were matched to the unknown and ranked by type as shown in Table 13. Additionally, it was possible to rank each individual tanker load from an oil field in a similar fashion. Typically, the average value of R between the unknown and SA04 load 133 is

$$R = \frac{0.0011785 + 0.000969 + \dots 0.008478}{7} = 0.006354$$

In this manner, several tanker loads from the same oil field can be ranked accordingly to R values. In fact Table 13 indicates correctly that load 133 is the proper match.

Method 1B correctly scored in all trials. Using the data generated from spill series A through E, all 16 mystery spill experiments were successfully matched to the correct oil field. In 14 out of 16 cases the next lowest residual, R, exceeded 100 percent and in 10 out of 16 exceeded 200 percent. An interesting result of method 1B, however, is the success in matching the unknown to the correct tanker load. In nine of the sixteen spills the type of crude oil spilled was present in the reference collection in multiple loads, and, in fact, as many as four. In all but two cases the correct tanker load was chosen as well as the correct type. In those two cases, the number of "unknown" replicates was small (three). Table 14 shows the summary of results for tagging method 1B.

During the course of the project, an actual spill of 1000 gallons of lagotreco occurred in Portland Harbor. Four replicate samples of the spill were collected and matched against a reference collection containing 15 oil types including the true material. Method 1B correctly selected the proper oil type and tanker load.

## METHOD 2: VISUAL COMPARISON OF CHROMATOGRAMS AND NUMERICAL COMPARISON OF V-N1 AND S-N RATIOS

A total of twelve oil spills were created out of series F,G, and O (four spills per series) to evaluate this method. Again, the products used were typical crude oils handled in the Portland area.

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TABLE 13. MINIMUM R VALUES, SPILL D METHOD 1B

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|    | Type<br>of Oil | Load<br>No | Number of<br>replicates | R        |
|----|----------------|------------|-------------------------|----------|
| 1  | SA04           |            | 4                       | 0.003997 |
|    |                | 133        | 1                       | 0.006355 |
|    |                | 120        | 1                       | 0.007487 |
|    |                | 115        | 1                       | 0.017374 |
|    |                | 125        | 1                       | 0.042030 |
| 2  | MX01           |            | 1                       | 0.029894 |
| 3  | PG02           |            | 1                       | 0.030167 |
| 4  | SA07           |            | 3                       | 0.030962 |
|    |                | 118        | 1                       | 0.030962 |
|    |                | 132        | 1                       | 0.101724 |
|    |                | 128        | 1                       | 0.115074 |
| 5  | SA05           |            | 2                       | 0.032348 |
|    |                | 111        | 1                       | 0.032348 |
|    |                | 127        | 1                       | 0.067668 |
| 6  | SA09           |            | 1                       | 0.034950 |
| 7  | SA11           |            | 1                       | 0.044544 |
| 8  | SA08           |            | 2                       | 0.045322 |
|    |                | 121        | 1                       | 0.045322 |
|    |                | 126        | 1                       | 0.074915 |
| 9  | PG11           |            | 1                       | 0.045722 |
| 10 | SA06           |            | 1                       | 0.076305 |
| 11 | SA01           |            | 1                       | 0.188195 |
| 12 | PG08           |            | 2                       | 0.320020 |
|    |                | 131        | 1                       | 0.320020 |
|    |                | 117        | 1                       | 0.411172 |
| 13 | A-01           |            | 1                       | 0.498037 |
| 14 | PG09           |            | 1                       | 0.532755 |
| 15 | SA03           |            | 1                       | 0.777349 |

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TABLE 14. SCORE SUMMARY, METHOD 1B

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| Trial<br>Spill | # in Ref.<br>Collection | FINAL CHOICE |        | CORRECT MATCH |        | # of Loads<br>per Type |
|----------------|-------------------------|--------------|--------|---------------|--------|------------------------|
|                |                         | Oil Type     | Load # | Oil Type      | Load # |                        |
| 1              | 6                       | SA07         | 30     | SA07          | 56     | 3                      |
| 2              | 9                       | PG08         | 106    | PG08          | 100    | 2                      |
| 3              | 9                       | SA04         | 99     | SA04          | 99     | 3                      |
| 4              | 9                       | SA04         | 99     | SA04          | 99     | 3                      |
| 5              | 15                      | SA09         | 112    | SA09          | 112    | 1                      |
| 6              | 15                      | SA05         | 111    | SA05          | 111    | 2                      |
| 7              | 15                      | PG02         | 113    | GP02          | 113    | 1                      |
| 8              | 15                      | PG08         | 131    | PG08          | 131    | 2                      |
| 9              | 15                      | SA04         | 120    | SA04          | 120    | 4                      |
| 10             | 15                      | SA04         | 133    | SA04          | 133    | 4                      |
| 11             | 15                      | SA07         | 118    | SA07          | 118    | 3                      |
| 12             | 15                      | SA07         | 132    | SA07          | 132    | 3                      |
| 13             | 12                      | PG06         | 135    | PG06          | 135    | 1                      |
| 14             | 12                      | SA01         | 134    | SA01          | 134    | 1                      |
| 15             | 12                      | SA06         | 136    | SA06          | 136    | 1                      |
| 16             | 12                      | A-02         | 137    | A-02          | 137    | 1                      |

SCORE (type) = 16/16 correct = 100 percent.

SCORE (load) = 7/9 correct = 78 percent.

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The evaluation process, by definition, must be subjective. Since there is no objective way to visually average replicate chromatograms, a single chromatogram of the unknown aged for 72 hours was used for comparison. Although selection of a 72 hour sample would be impossible in an actual spill, it was necessary to establish such a criteria because crudes subjected to weathering longer than a week became increasingly difficult to analyze visually. Therefore, the success rate of this method (Method 2) is artificially high.

A panel of three independent judges was used to evaluate. The panel first made visual comparisons between low resolution chromatograms of the unknown and reference sets. Based on an "unlike" or "like" decision, a representative (or representatives) from the reference collection were selected for further comparison. Again the panel judged the goodness of fit between high resolution chromatograms of the unknown and a reference collection reduced according to the preliminary screening. Finally, the panel used V/Ni and S/N ratios in its matching assessment. To facilitate comparison of the element ratios, an overall ratio can be calculated:

$$R_V = \frac{(V/Ni)_u}{(V/Ni)_k} \text{ and } R_S = \frac{(S/N)_u}{(S/N)_k}$$

Where (V/Ni) = ratio of V to Ni concentrations,

(S/N) = ratio of S to N concentrations,

and u, k = unknown and known respectively.

For an ideal match the result would be unity. Conversely, the greater the mismatch, the greater the deviation from unity. Table 15 gives an example of element ratio data from one trial in spill series F. The table clearly distinguishes between the Persian Gulf crudes (PG series) and the South American crudes (SA series). In fact, in this case the correct match can be made on the assessments of element ratios alone. Although not done for this study, the element ratios can advantageously be treated in the same manner as the chromatogram ratios to develop a residual value R as previously described.

Results of passive tagging by Method 2 are shown in Table 16. By this method seven out of twelve unknowns were correctly identified for a score of 58 percent. In three of the unsuccessful cases, the correct oil type was one of two choices, but no clear decision could be made as to correct assignment in the opinion of the judges. In the remaining two unsuccessful cases, wrong choices were clearly made. Matching decisions by this method can be only

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TABLE 15.  $R_v$  and  $R_s$ , SPILL F METHOD 2

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|         | <u>(V/N1)</u> | <u>(S/N)</u> | <u>Rv</u> | <u>Rs</u> |
|---------|---------------|--------------|-----------|-----------|
| unknown | 5.2619        | 26.4698      |           |           |
| PG02    | 3.6887        | 9.7273       | 1.42650   | 2.72118   |
| PG04    | 3.8846        | 22.5556      | 1.35457   | 1.17353   |
| (PG05)  | 4.5705        | 20.0857      | 1.15128   | 1.31784   |
| PG06    | 3.9552        | 18.3158      | 1.33039   | 1.44519   |
| PG08    | 3.4202        | 15.4317      | 1.53849   | 1.71528   |
| PG09    | 3.3586        | 23.2353      | 1.56671   | 1.13920   |
| PG10    | No Results    |              |           |           |
| PG11    | 2.0016        | 12.8984      | 2.62887   | 2.05217   |
| SA04    | 8.7349        | 7.7018       | 0.60240   | 3.43683   |
| SA05    | 11.3546       | 9.5231       | 0.46342   | 2.77953   |
| SA06    | 11.4253       | 7.6522       | 0.46055   | 3.45910   |
| SA07    | 8.4931        | 7.7182       | 0.61956   | 3.42953   |
| SA08    | 9.4204        | 6.9257       | 0.55857   | 3.82196   |
| SA09    | 8.7205        | 7.3228       | 0.60340   | 3.61470   |
| SA10    | 8.3427        | 7.2832       | 0.63072   | 3.63436   |

— = correct match

( ) = best fit

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TABLE 16. SCORE SUMMARY, METHOD 2

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| Trial<br>(Spill) | No. in<br>Reference<br>Collection | Possible Choices<br>Following Preliminary<br>GC Sort and<br>Use of Rv and<br>Rs Ratios |  | Final<br>Choice      | Correct<br>Match |
|------------------|-----------------------------------|----------------------------------------------------------------------------------------|--|----------------------|------------------|
|                  |                                   |                                                                                        |  |                      |                  |
| 1                | 15                                | PG08<br>PG09                                                                           |  | PG08                 | PG09             |
| 2                | 15                                | PG05                                                                                   |  | PG05                 | PG05             |
| 3                | 15                                | SA10<br>PG04                                                                           |  | SA10                 | SA10             |
| 4                | 15                                | PG10<br>PG11                                                                           |  | Indecisive<br>Result | PG11             |
| 5                | 8                                 | SA04<br>SA05<br>SA07<br>SA08                                                           |  | SA05                 | SA05             |
| 6                | 8                                 | SA04<br>SA05<br>SA07<br>SA08                                                           |  | Indecisive<br>Result | SA08             |
| 7                | 8                                 | PG11                                                                                   |  | PG11                 | PG11             |
| 8                | 8                                 | PG06<br>PG09                                                                           |  | PG09                 | PG09             |
| 9                | 8                                 | PG02                                                                                   |  | PG02                 | PG02             |
| 10               | 8                                 | SA05<br>SA07<br>SA12                                                                   |  | SA05                 | SA07             |
| 11               | 8                                 | SA05<br>SA06                                                                           |  | Indecisive<br>Result | SA05             |
| 12               | 8                                 | NA01<br>PG02                                                                           |  | NA01                 | NA01             |

Score = 7/12 correct = 58 percent

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carried to the oil field designation at best with no likelihood of discriminating between different tanker loads from the same oil field or other similar sources. In light of the fact that, as described at the outset, the experiments were designed to give an artificially high success rate plus the fact that measured success rate was low (58 percent), Method 2 does not seem to hold much promise. Apparently the subjective evaluation of chromatograms and possibly, loss in chromatogram detail due to high temperature stripping results in a fatal loss of discriminatory power.

METHOD 3: NUMERICAL COMPARISON OF V-NI AND S-N AND NUMERICAL COMPARISON OF HIGH RESOLUTION GAS CHROMATOGRAPHY

This method combines the best of Method 1 (Numerical Comparison of High Resolution Chromatograms) with the best of Method 2 (Comparison of V-Ni and S-N ratios). To evaluate this method, data from spill series F,G, and O used in Method 2 were recalculated. In addition, two more spill series (H and P) were evaluated. The series H and P were prepared essentially according to Method 2 except that series P was stripping with nitrogen gas at 200 C. Also, only V/Ni data were available for spill series P. In all, 20 spills were matched using 5 spill series.

Samples of the unknown and reference collection were prepared for high resolution chromatography and element analysis according to Method 2. Chromatogram residual (R) data by Method 1B and element ratios ( $R_v$  and  $R_s$ ) were calculated. Table 17 shows a typical data display.

Results of matching spills series F,G,O,H, and P are listed in Table 18. A success rate of only 70 percent was observed. Perhaps more indicative of the reduced accuracy was the fact that, although no errors were made, six indecisive results was recorded. Using the less successful Method 1B, an even poorer success rate of 65 percent was determined. It would appear that the high temperature (280°C) stripping procedure employed in preparing these samples significantly alters concentrations in the  $C_{17}$  to  $C_{20}$  range to effectively reduce differences between oils. A further indication that the loss in resolving power is related to stripping temperature is the result of series P. In this case, stripping occurred at 200°C, not 280°C, and all four spills were correctly identified. This is the only spill series in F,G,O,H, and P in which all four unknowns were correctly identified, even marginally.

It appears that Method 3 has the best chances for success. Since it combines Method 1 (83+ percent success) with Method 2 (70 percent success, representing less than optimum conditions due to high temperature stripping) then one might reasonably expect a success rate substantially higher than 83 percent under optimum conditions.

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TABLE 17. TYPICAL DATA FOR METHOD 3

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|   | I.D. | Number of<br>replicates | R        | Rv     | Rs     |
|---|------|-------------------------|----------|--------|--------|
| 1 | SA08 | 4                       | 0.045512 | 1.5259 | 1.0244 |
|   | 301  | 1                       | 0.048542 |        |        |
|   | 318  | 1                       | 0.048950 |        |        |
|   | 305  | 1                       | 0.049608 |        |        |
|   | 330  | 1                       | 0.050124 |        |        |
| 2 | PG11 | 1                       | 0.051386 | 7.1817 | 0.5500 |
| 3 | PG02 | 1                       | 0.054547 | 3.8970 | 0.7293 |
| 4 | PG04 | 4                       | 0.057771 | 1.6457 | 0.9211 |
|   | 331  | 1                       | 0.058969 |        |        |
|   | 316  | 1                       | 0.059689 |        |        |
|   | 326  | 1                       | 0.060429 |        |        |
|   | 327  | 1                       | 0.063472 |        |        |
| 5 | SA05 | 5                       | 0.059633 | 1.2660 | 0.7450 |
|   | 342  | 1                       | 0.063859 |        |        |
|   | 319  | 1                       | 0.080403 |        |        |
|   | 329  | 1                       | 0.082829 |        |        |
|   | 297  | 1                       | 0.084393 |        |        |
|   | 333  | 1                       | 0.085676 |        |        |
| 6 | SA07 | 4                       | 0.061020 | 1.6925 | 0.9192 |
|   | 296  | 1                       | 0.065101 |        |        |
|   | 320  | 1                       | 0.065747 |        |        |
|   | 328  | 1                       | 0.077411 |        |        |
|   | 332  | 1                       | 0.079780 |        |        |
| 7 | PG13 | 3                       | 0.288843 | 3.7992 | 0.4534 |
|   | 300  | 1                       | 0.288843 |        |        |
|   | 335  | 1                       | 0.350392 |        |        |
|   | 340  | 1                       | 0.355139 |        |        |
| 8 | PG09 | 1                       | 0.501903 | 4.2800 | 0.3053 |

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TABLE 18. SCORE SUMMARY, METHOD 3

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| <u>Trial<br/>(Spill)</u> | <u>No. in<br/>Reference<br/>Collection</u> | <u>Possible choices<br/>Following GC Sort</u> | <u>Final Choice<br/>Based on<br/>Element Data</u> | <u>Correct<br/>Match</u> |
|--------------------------|--------------------------------------------|-----------------------------------------------|---------------------------------------------------|--------------------------|
| 1                        | 15                                         | PG08.<br>PG09                                 | PG09                                              | PG09                     |
| 2                        | 15                                         | PG05                                          | PG05                                              | PG05                     |
| 3                        | 15                                         | SA10                                          | SA10                                              | SA10                     |
| 4                        | 15                                         | SA06<br>SA08<br>SA09<br>PG02<br>PG10<br>PG11  | Indecisive<br>Result                              | PG11                     |
| 5                        | 8                                          | SA05<br>SA07                                  | SA05                                              | SA05                     |
| 6                        | 8                                          | SA08<br>PG11<br>PG02<br>SA04                  | SA08                                              | SA08                     |
| 7                        | 8                                          | SA04<br>SA05<br>SA08<br>PG02<br>PG11          | Indecisive<br>Result                              | PG11                     |
| 8                        | 8                                          | PG09                                          | PG09                                              | PG09                     |
| 9                        | 8                                          | PG11<br>PG02                                  | PG02                                              | PG02                     |
| 10                       | 8                                          | SA07                                          | SA07                                              | SA07                     |
| 11                       | 8                                          | SA05<br>SA06<br>PG02                          | SA05                                              | SA05                     |
| 12                       | 8                                          | NA01                                          | NA01                                              | NA01                     |

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| <u>Trial<br/>(Spill)</u> | <u>No. in<br/>Reference<br/>Collection</u> | <u>Possible choices<br/>Following GC Sort</u> | <u>Final Choice<br/>Based on<br/>Element Data</u> | <u>Correct<br/>Match</u> |
|--------------------------|--------------------------------------------|-----------------------------------------------|---------------------------------------------------|--------------------------|
| 13                       | 18                                         | PG05<br>PG13                                  | Indecisive<br>Result                              | PG13                     |
| 14                       | 18                                         | SA05<br>PG11<br>SA04<br>PG03<br>PG02          | Indecisive<br>Result                              | PG02                     |
| 15                       | 18                                         | SA07<br>PG03<br>SA04<br>PG02                  | Indecisive<br>Result                              | SA07                     |
| 16                       | 18                                         | PG04<br>PG05<br>PG06<br>PG13                  | Indecisive<br>Result                              | PG04                     |
| 17                       | 33                                         | SA13<br>SA05<br>SA06<br>PG03<br>NA01          | SA13                                              | SA13                     |
| 18                       | 33                                         | SA02<br>SA13                                  | SA02                                              | SA02                     |
| 19                       | 33                                         | PG05                                          | PG05                                              | PG05                     |
| 20                       | 33                                         | NA01<br>SA06<br>SA05                          | NA01                                              | NA01                     |

Overall Score = 14/20 Correct = 70 Percent  
Trial 17, 18, 19, 20 Score = 4/4 Correct = 100 Percent

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METHOD 4: VISUAL COMPARISONS OF INFRARED SPECTRA

Typical spectra of several crudes are shown in illustration 9. Of particular concern during this study was the influence of weathering on the spectral pattern. The absorption of this highly complex mixture undoubtedly results from group frequencies and not from the fingerprint skeletal vibrations. The major contributions to the intensity of the pattern will be made by those components present in the oil in highest concentration. It would seem, therefore, that the bands in question arise predominantly from low molecular weight aromatic compounds present in crude oil. Although these aromatic compounds represent a particularly stable group of materials in crude oil, the low molecular weight molecules possess considerable solubility and volatility. Therefore, it was not completely unexpected to observe that the entire pattern washed out of samples weathered for more than 24 hours, rendering the technique of limited passive tagging value. It appears that infrared passive tagging in the  $600$  to  $400\text{ cm}^{-1}$  region holds little hope as a passive tagging technique.

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WEATHERING EVALUATIONS

The assumption was made during tagging evaluations that weathering would not effect the relative concentrations of the four hydrocarbons involved in the analysis. In order to test this assumption the entire data file for Spills C-G and O has been recalculated by Method 1B dropping first the 316 hour sample and then both the 316 and 148 hour samples. Dropping the 316 hour sample measurably improved the correct residual values in twelve of twenty-two cases. In nine cases dropping the most heavily aged sample collected caused no change or resulted in a slight increase in the value. In only one case did the residual value measurably degrade. When both the 148 hour and the 316 hour samples were dropped the number of improved residuals was cut by 25 percent and the number of degraded residuals rose by a factor of 5 indicating that no real advantage was gained in removing that additional data point.

These results seem to indicate that degradation of R values do occur within some oils during the two week period, but that measurable changes do not develop until after the first week. If unknown samples were collected only during the second week of a spill it would appear that some difficulty might be experienced in identifying the correct source oil from closely matching neighbors. Table 19 shows passive tagging residual values (R) as a function of sample age class removed from the calculation. Results of sample age class removal are summarized in Table 20.

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TABLE 19. PASSIVE TAGGING R VALUE CHANGES AS A FUNCTION OF SAMPLE AGE

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Maximum Age Class Contained in Residual Calculation

| Spill # | 316 hrs. |      | 148 hrs.<br>(1) |      | 76 hrs.<br>(1)  |      |
|---------|----------|------|-----------------|------|-----------------|------|
|         | R-Value  | Rank | R-Value ( %)    | Rank | R-Value ( %)    | Rank |
| C-1     | 0.001962 | 1st  | 0.000683 ( 65)  | 1st  | 0.000683 ( 0)   | 1st  |
| D-1     | 0.012256 | 1st  | 0.013637 (-11)  | 1st  | 0.015212 ( -12) | 2nd  |
| D-2     | 0.068261 | 1st  | 0.060317 ( 12)  | 1st  | 0.009629 ( 84)  | 1st  |
| D-3     | 0.007420 | 1st  | 0.003367 ( 55)  | 1st  | 0.001696 ( 50)  | 1st  |
| D-4     | 0.003997 | 1st  | 0.003250 ( 19)  | 1st  | 0.003300 ( -2)  | 1st  |
| D-5     | 0.006740 | 1st  | 0.002804 ( 58)  | 1st  | 0.001202 ( 57)  | 1st  |
| E-1     | 0.056410 | 1st  | 0.004664 ( -8)  | 1st  | 0.071391 ( -17) | 1st  |
| E-2     | 0.003242 | 1st  | 0.001415 ( 56)  | 1st  | 0.001433 ( -1)  | 1st  |
| E-3     | 0.023949 | 1st  | 0.027621 (-15)  | 1st  | 0.031530 ( -14) | 1st  |
| E-4     | 0.010360 | 1st  | 0.009775 ( 6)   | 1st  | 0.011236 ( -15) | 1st  |
| F-1     | 0.030005 | 2nd  | 0.023533 (-12)  | 2nd  | 0.036398 ( -55) | 2nd  |
| F-2     | 0.025798 | 1st  | 0.017285 ( 33)  | 1st  | 0.017314 ( 0)   | 1st  |
| F-3     | 0.036000 | 1st  | 0.019626 ( 46)  | 1st  | 0.010380 ( 47)  | 1st  |
| F-4     | 0.035709 | 4th  | 0.025553 ( 28)  | 3rd  | 0.010224 ( 60)  | 2nd  |
| G-1     | 0.045564 | 1st  | 0.008377 ( 82)  | 1st  | 0.001459 ( 83)  | 1st  |
| G-2     | 0.045512 | 1st  | 0.006395 ( 86)  | 1st  | 0.003034 ( 53)  | 1st  |
| G-3     | 0.059486 | 3rd  | 0.012923 ( 78)  | 2nd  | 0.006595 ( 49)  | 2nd  |
| G-4     | 0.008497 | 1st  | 0.006528 ( 23)  | 1st  | 0.004499 ( 31)  | 1st  |
| O-1     | 0.009605 | 2nd  | 0.003737 ( 13)  | 2nd  | 0.010671 (-186) | 2nd  |
| O-2     | 0.006399 | 1st  | 0.006905 ( -8)  | 1st  | 0.006184 ( 10)  | 1st  |
| O-3     | 0.004166 | 1st  | 0.004908 (-18)  | 1st  | 0.005760 ( -17) | 2nd  |
| O-4     | 0.002016 | 1st  | 0.002103 ( -4)  | 1st  | 0.002580 ( -23) | 1st  |

(1)

Positive % corresponds to a reduction in R-Values and a Negative % sign indicates an increase in R-Values.

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TABLE 20. SUMMARY OF SAMPLE AGE CLASS ON R

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Maximum Age Class Contained in Residual Calculation

|                                     | <u>148 hrs.</u> | <u>76 hrs.</u> |
|-------------------------------------|-----------------|----------------|
| Residual Values Improved            | 12              | 9              |
| Residual Values Remain the Same (1) | 9               | 8              |
| Residual Values Degraded            | 1               | 5              |

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(1) Excursions of  $\pm 15\%$  are arbitrarily considered to represent essentially an unchanged value.

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SECTION 8

QUALITY CONTROL

SCOT column performance was important. Since the demand approached 200-300 chromatograms per column, the continuing ability of a column to successfully and reproducibly resolve C-17, Pristane, C-18, and Phytane was critical, especially for Method 1 where numerical evaluation was required. To monitor column performance, one sample of Orito crude was selected as a standard. The crude was desulfured according to Method 1 procedures and the light ends stripped at 200°C with nitrogen. The Orito standard was stored at -20°C until needed.

Chromatograms of the standard were run periodically. Typically, the standard chromatogram was run at least once each day. On the average, the standard was chromatogrammed about once for every 8 samples (12%). In this manner, both intercolumn and intracolumn performance was monitored. Table 21 shows the results of the Orito standard run on all columns. Reproducibility of the ratios is very good. In the worst case, a variance of 4.2 percent was experienced. Therefore, intercolumn and intracolumn comparisons of chromatographic data should be readily achievable.

During the spill Series D, an accidental spill of several thousand gallons of Lagotrec crude oil (SA07) occurred in Portland Harbor during an unloading operation. Because the incident happened before the start of the spill series, it was possible to duplicate the actual spill under experimental conditions. The calculated residuals by Method 1B against a reference file of 23 crudes compared extremely well:  $R = 0.006442$  and  $R = 0.006740$  for the experimental and actual spills respectively.

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TABLE 21. COLUMN PERFORMANCE

| Column No. | Total<br>Chromatograms | Number of<br>Standards | R1  |     | R2  |     | R3  |     | R4  |     |
|------------|------------------------|------------------------|-----|-----|-----|-----|-----|-----|-----|-----|
|            |                        |                        | M   | SD  | M   | SD  | M   | SD  | M   | SD  |
| 18         | 143+                   | 14                     | 1.2 | 2.5 | 1.2 | 3.9 | 1.2 | 3.4 | 1.1 | 2.5 |
| 20         | 69+                    | 11                     | 1.2 | 2.9 | 1.2 | 2.4 | 1.1 | 2.3 | 1.2 | 1.9 |
| 21         | 148+                   | 18                     | 1.1 | 3.1 | 1.2 | 3.0 | 1.1 | 1.9 | 1.1 | 1.6 |
| 22         | 67+                    | 12                     | 1.2 | 3.5 | 1.3 | 4.2 | 1.2 | 3.3 | 1.1 | 3.0 |
| 23         | 77+                    | 15                     | 1.2 | 2.3 | 1.1 | 1.2 | 1.1 | 2.1 | 1.2 | 1.7 |

NOTE: M = Mean  
SD = Standard Deviation (% relative)  
All Columns are 15 meter SCOT, OV1 Capillary  
R1 = heptadecane/pristane  
R2 = heptadecane/octadecane  
R3 = pristane/phytane  
R4 = octadecane/phytane

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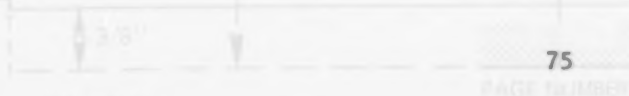
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